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PROCESS FOR THE PRODUCTION OF MELTS FOR THE  
GENERATION OF PROPELLANTS

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Assignee: Wolff & Co., Inc., Walsrode (Hannover)

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PROCESS FOR THE PRODUCTION OF MELTS FOR THE  
GENERATION OF PROPELLANTS

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ABSTRACT. Disadvantages of hitherto used methods are shown. Object of invention is a new process of obtaining propellants which involves less cost, time and danger, and furnishes a better end product. In this a mixture of nitrocellulose and a nitrated polyalcohol, containing at least 10% water and produced at a temperature below 18°C, is fed into a molten nitro compound.

In the case of manufacture of propellants such as cannon- and gunpowder /1\* and rocket compositions, one has hitherto proceeded in such a manner that a mixture of nitrocellulose, nitroglycerine or one or several similar nitrated multivalent alcohols and water is dehydrated on heated rolls and that the nitrated multivalent alcohols thus bring about gelatinization of the nitrocellulose. The gelatinized mixtures, rolled out into thin skins, are then converted into strip or foil shape with the aid of a cutting device, while for the production of tubular powder the desired shape is imparted to the gelatinized mass by means of a press with an appropriately shaped nozzle.

This process for the manufacture of propellants has the disadvantage that the rolling work for the dehydration of powder mixtures not only consumes much time and energy but also involves considerable danger, since at a higher temperature the masses are sensitive to friction and therefore ignite easily. Moreover, the vapors developing during the rolling process of the nitrated alcohols are a severe burden to persons operating the rolling mill.

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\* Numbers in the margin indicate pagination in the foreign text.

In the field of the production of explosives, i.e., of quickly reacting explodable mixtures of chemical compounds accompanied by a gas development with crushing and blasting effects, it has already been possible to avoid in a simple manner the drawbacks connected with the dehydration of the mixture to be processed by rolling in such a way that the explosive components are mixed in a molten state and this melt is then poured into molds, for instance for grenades and bombs. Transfer of this work principle to the production of propellants, i.e., of slowly reacting explodable mixtures of chemical compounds accompanied by gas development which expells a projectile from gun, cannon or rocket tubes, encounters difficulties in practice, especially in the case of propellant charges formed with nitrocellulose and nitrated multivalent alcohols, since during introduction of the water-containing mixture which is prepared in the conventional manner, for instance nitrocellulose and diglycoldinitrate, into molten trinitrotoluol or into another molten nitro substance, only part of the nitrocellulose gelatinizes, without the desired homogeneous solution being formed. /2

For the manufacture of propellant powders with delayed initial combustion speed it has also already been proposed to convert into a solution a restraining substance, such as diamyl or dibutylphthalate or mono-, di-, or trinitrotoluol, in methyl or ethyl alcohol or benzene, and to mix with this solution the mass of grains of powder, for instance of nitrocellulose-nitroglycerine powder, for the purpose of superficial treatment with the addition of water, and then to heat the mixture to bring about the evaporation of the solvent and the water. This process works with a dissolved mixture with subsequent elimination and recovery of the solvent from it, and is therefore cumbersome and expensive.

It has been shown on the basis of exhaustive experiments that in the case of production of molten powder mixtures containing nitrocellulose and nitrated multivalent alcohols as well as nitro compounds, the attainment of homogeneous solutions prerequisites that, while it is being fed into the molten nitro compounds, the nitrocellulose enriched by the nitrated multivalent alcohols have a very special water content which amounts at least to 18%, preferably 20 to 23%, since in the case of too low a water content the nitrocellulose does not completely go into solution and small lumps remain which have a very unfavorable effect upon the ballistics of the powder, and too high a water content results in too long a period for the melting process which is disadvantageous for the stability of the final product.

The experiments have furthermore shown that the temperature to be maintained during the mixing of nitrocellulose with the nitrated multivalent alcohol, in order to prevent the start of gelatinization prior to the feeding of the nitrocellulose into the melt and to avoid the undesirable formation of small lumps occurring at excessive temperatures, must be fixed as low as possible and satisfies this condition only if it never exceeds 18°C.

On the basis of these findings, in accordance with the invention, the process for the production of propellants is such that a mixture of nitrocellulose and a nitrated polyalcohol, containing at least 10% water and being

produced at a temperature of below  $10^{\circ}\text{C}$ , is fed into a molten nitro compound. In this way not only the rolling work usually required in a case of production of propellants for the dehydration of powder mixture is avoided, but also an easily pourable, completely uniform melt without any lump formation is attained. Also according to the invention, no solvent is employed, which simplifies the entire process and makes it less expensive.

Here it is advantageous to use a nitrocellulose with a nitrogen content below 12.4% since a nitrocellulose containing more nitrogen, as has been shown, yields melts which even in the case of low viscosity of the nitrocellulose are highly viscous, and conversion into the desired form and the removal of air is therefore impossible or difficult. It has furthermore been found advantageous to conduct the evaporation of water during the dissolution process at such a speed as to bring about complete dissolution of the nitrocellulose in the melt.

Particularly suited for the nitro substance, which in a molten state serves to dissolve the nitrocellulose admixed with a nitrated polyalcohol, and the melting point of which must therefore lie below the decomposition temperature of the nitrocellulose, i.e., below  $130^{\circ}\text{C}$ , are aromatic and aliphatic nitro compounds. Di- or trinitrotoluol, di- or trinitrobenzene, di- or trinitrophenol, di- or trinitrophenyl glycol ether nitrate, tetranitromethyl aniline, mononitronaphthalene, trinitronaphthalene, dinitroxytoluol, pentanitrodiphenylamine, trinitrophenylglycine, trinitrophenylethanolamine are, for instance, employed as aromatic nitro substances, and dinitrodiethanol nitratoxamide, tetranitropenterytrite, dioxyethylsulfodinitrate are used as aliphatic nitro substances. According to the invention, mixtures of these aromatic and aliphatic nitro substances and of these nitrates can also be used for gelatinization of the nitrocellulose. Nitroglycerine, diglycoldinitrate and similar alcohol nitrates are, for instance, suitable as nitrated multivalent alcohols. Part of the nitrated multivalent alcohol to be mixed with the nitrocellulose can also be replaced by softeners.

For practical carrying out of the process according to the invention it has furthermore been proved advantageous to employ a nitrocellulose, the viscosity of which has been reduced by autoclave treatment. The pressure cooking is conducted under such conditions that the viscosity amounts to 15 to 25 seconds. The viscosity is here determined in such a way that a 10% solution of the nitrocellulose to be measured is measured in a mixture of butanol, butyl acetate and toluol at a ratio of 3:4:5 in a Cochius viscosimeter (7 mm) at  $18^{\circ}\text{C}$ . /3

#### Example

53 kg trinitrotoluol are heated in a cauldron equipped with a heating jacket, and in addition 28 kg nitrocellulose with a nitrogen content of 12.1% and a viscosity of 20 seconds, measured in 3% acetic solution according to Cochius, are intimately mixed at  $14^{\circ}\text{C}$  with 18 kg diglycol dinitrate in aqueous emulsion. This mixture is relieved of excessive water in a centrifuge to such an extent that it still possesses a humidity of 22%. The nitrocellulose which has thus

been enriched with nitrated multivalent alcohol is now prepulverized in the conventional manner and then is slowly stirred into the molten trinitrotoluol, with very thorough mixing. Thereupon the melting cauldron is sealed and put under vacuum, so that the water gradually evaporates. The dehydration process must here be controlled in such a way that gelatinization proceeds in parallel with the evaporization, since too fast an evaporization of the water hinders the the gelatinization. The yielded homogeneous, easily pourable melt is converted in the conventional manner into the desired powder.

#### Patent Claims

1. Process for the production of melts for the production of propellants, characterized by the fact that a mixture of nitrocellulose and a nitrated polyalcohol, produced at a temperature of 18°C and containing at least 18% water, is fed into a molten nitro compound.

2. Process according to Claim 1, characterized by the fact that a nitrocellulose with a nitrogen content below 12.4% is employed.

3. Process according to Claims 1 and 2, characterized by the fact that nitrocellulose which has undergone viscosity reduction by means of autoclave treatment, is employed.

4. Process according to one of Claims 1 to 3, characterized by the fact that the evaporization of water during the dissolution process is carried out at such speed that complete dissolution of the nitrocellulose takes place in the melt.

5. Process according to one of Claims 1 to 4, characterized by the fact that a part of the nitrated multivalent alcohol to be mixed with the nitrocellulose is replaced by softeners.

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Printed works considered: USA Patent Specification No. 1 967 913.

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